| DATE: | 5/17/82 |
|---------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| SUBJECT: | Review of Region V Contractor Data; Received for Review on 5/k/82 |
| | |
| FROM: | Curtis Ross, Director (Central Regional Laboratory |
| · TO : | Data User: TAT, CDO, HolosKA |
| | We have reviewed the data for the following Case(s): Site Name: SAUSET SMO Case No: Y905 EPA Data Set No: SF140/ Decision Unit: CRL No's: & 2 W TO 6 SU/ Hev 82 W TO 6 SU/ SMO Traffic No.'s: |
| | Contractor Lab: WRight State V. Person-hours required for review: |
| | Following are our findings: |
| | note Damples were preserved correctly, but detection limits for this study were higher than the previous study - accounting for the differences in compounds seen/not seen. |
| · | MAK |
| | |
| ~ | |
| | Data are unacceptable for use. But fails to meet program objective Data are unacceptable for use. |
| | () Data are preliminary - this case has been forwarded to Dr. Alfred Haeberer, EPA Support Services, for review - pending reply. |
| | cc: Dr. Alfred Haeberer, EPA Support Services |

EPA FORM 1320-6 (RFV. 3-76

WRIGHT STATE

May 3, 1982

Wright State University Dayton, Ohio 45435

> Mr. Curtis Ross United States Environmental Protection Agency Region V 230 S. Dearborn Chicago, Illinois 60604

RE: EPA Order No. 56606 NAEX

Dear Mr. Ross:

Saught dudy | 42)
Brehm Laboratory
513/873-2202

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US EPA CONTACT REGIONAL LAB. 536 S. CLACK STARST CHICAGO, ILL HOLD COULD

All analyses specified under Tasks 1 and 2 of the subject EPA Purchase Order No. 56606 NAEX have now been completed by our laboratory. As you know, each of the five water/sediment samples were analyzed for CDDs/CDFs as required under Task 1 and these data, as well as a complete description of the analytical methodology employed, were formally transmitted to you in an interim report dated March 16, 1982. Regarding our telephone conversation of March 30, 1982 in which you inquired about precursors of chlorinated dibenzo-p-dioxins (CDDs) which could possibly be present in the Sauget Landfill, it should be emphasized that various compounds are known which are precursors for the CDDs. For example, chlorinated phenoxyphenols, chlorinated phenols, chlorinated benzenes and possibly even polyvinyl chloride polymers have, under certain conditions, been found to give rise to CDDs. In addition, CDDs have been detected in stack effluents arising from municipal waste incineration. Regarding the question of whether or not precursors such as the chlorophenoxyphenols, if present in the environmental sample, could, under conditions of analysis undergo dehydrohalogenation and give rise to CDDs, we feel that if phenoxyphenols were present at concentrations comparable to the concentrations of CDDs which were found in the samples, that the sample clean-up methodology would effectively remove these prior to gas chromatographic-mass spectrometric analysis. The presence of large concentrations of phenoxyphenols (perhaps 100% concentration of CDDs in the sample) could conceivably overwhelm the sample clean-up procedure, but, no specific evidence exists which indicates that large concentrations of phenoxyphenols do indeed generate CDDs during analysis. The phenoxyphenol question should be studied further, but this is difficult at present since well-characterized standards are not readily available. If environmental samples do contain chlorinated phenoxyphenols, it is possible that, under certain conditions which could exist in a chemical landfill, cyclization of these compounds could occur and give rise to CDDs. Here again experimentation is required in order to substantiate this possibility.

The purpose of the present report is to summarize the methodology employed and the results obtained in assaying the five water/sediment samples for the various compounds specified by EPA under Task 2 of the subject purchase order. The samples received for analysis at the beginning of the project are listed in Table 1 and the descriptions listed therein are based upon observations made in this laboratory at the time of receipt of samples. Table 2 lists the organic compounds which were to be determined under Task 2 of the EPA order.

Mr. Curtis Ross May 3, 1982 Page 2

Obviously, several different isomers are possible for some of the compounds listed by EPA, and in these cases, calibrations were accomplished using representative isomers of these compounds, but not all possible isomers. The representative compounds used for calibration and quality assurance purposes are also listed in Table 2. High Performance Liquid Chromatography (HPLC) was employed to detect and quantitate the compounds of interest which were present in extracts of each of the water/sediment samples. The details of the analytical methodology employed are given in the Analytical Protocol appended to this report. The analytical results obtained are discussed below.

Initially, the methodology was verified by accomplishing analyses of standard solutions and when satisfactory results were obtained, actual samples were analyzed along with actual samples which had been spiked with the compounds of interest. Copies of representative chromatograms are attached as Figures 1-7. The data obtained are also listed in tabular form in Table 3. As seen in Table 3, recoveries of the compounds from actual samples prepared to contain known concentrations of the compounds of interest were satisfactory. However, the water/sediment samples themselves were found to contain no detectable levels of the pertinent compounds. These data are not in agreement with the results obtained previously by EPA, which were appended to the EPA order received by Wright State. The concentrations of the pollutants listed by EPA as being detected in similar samples are on the order of 5-10 times the minimum detectable concentrations achieved in the present analyses. The results obtained in the present analyses, therefore, may indicate that the water samples were not adequately preserved at the time of sampling. If appropriate reagents were not added to the water samples at the time of sampling (see, for example, the attached recommendations from Standard Methods For Water and Wastewater Analysis) then microbial degradation of some, if not all, of the compounds of interest could have occurred prior to analysis. The apparent absence of appreciable concentrations of both the pollutants of interest and of any similar compounds tends to further suggest that some degradation of the organic compounds may have occurred. Further analyses of fresh samples (with added preservatives) would indicate whether or not the lack of preservation was a problem with the present samples.

This completes this work called for under EPA Order No. 56606 NAEX. Our invoice is being submitted under separate cover. If you have any questions or comments regarding these data, please don't hesitate to call us. We appreciate this opportunity to work with USEPA on this important project.

Sincerely,

Thomas O. Tiernan, Ph.D. Professor of Chemistry and Director of Brehm Laboratory

Michael L. Taylor, Ph.D. Associate Professor of Pharmacology/Toxicology and Associate Director of

Brehm Laboratory

TABLE 1

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435

LISTING OF SAMPLES RECEIVED FROM USEPA (CHICAGO, REGION V) 1.

| EPA I.D. No. | WSU Sample No. | Description |
|-----------------|----------------|------------------------------|
| E1205 82WT06S01 | CWS-1 | 1 gallon of water/sediment |
| E1206 82WT06S03 | CWS-2 | 3/4 gallon of water/sediment |
| E1208 82WT06S05 | CWS-3 | 1 gallon of water/sediment |
| E1207 82WT06S07 | CWS-4 | 3/4 gallon of water/sediment |
| 82WT06R01 | CWS-5 | 3/4 gallon of water/sediment |

^{1.} Samples were received on January 14, 1982. Samples were packed in styrofoam beads, and ice water was present in shipping containers. Samples CWS-2 and CWS-5 were shipped together in one container and samples CWS-1,-3 and -4 were shipped together in a second container. Caps on bottles were taped.

TABLE 2

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435 SUSPECTED POLLUTANTS AND REPRESENTATIVE COMPOUNDS ANALYZED UNDER TASK #2, EPA

ORDER 56606 NAEX

| Con | npound | ds | Listed |
|-----|--------|----|--------|
| in | Task | #2 | 2 |

- 1. Chloroaniline
- 2. Chloronitrobenzene
- 3. Dichlorophenol
- 4. 2,4-D
- 5. Phenol
- 6. Methylbenzosulfaamide
- 7. Benzoic Acid
- 8. Benzene carboxylic acid
- 9. Dichloraniline

Representative Compounds Employed in Calibration/QC Studies

3-Chloroaniline

1-Chloro-2-nitrobenzene

2,4-dichlorophenol

2,4-dichlorophenoxyacetic acid

phenol

p-toluenesulfonamide

benzoic acid

3,5-dichloroaniline

TABLE 3

BREHM LABORATORY, WRIGHT STATE UNIVERSITY, DAYTON, OHIO 45435

ANALYTICAL RESULTS OBTAINED FOR SUSPECTED POLLUTANTS AND REPRESENTATIVE COMPOUNDS

| | WSU Sample No. 1 | | | | Spiked CWS-2 | Spiked CWS-3 | |
|-------------------------------------------------|------------------|-------|-------|-------|--------------|---------------------|---------------|
| Suspected Pollutant | CWS-1 | CWS-2 | CWS-3 | CWS-4 | CWS-5 | Found (added) ng/ml | Found (added) |
| Chloroaniline. | ND | ND | ND | ND | ND | | 903(1000) |
| Chloronitrobenzene | ND. | ND | ND | ND | ND | | 3,500(5,090) |
| Dichlorophenol | ND | ND | ND | ND | ND | 900(1030) | |
| 2,4-D | ND | ND | ND | ND | ND | 10,000(11,000) | May diff |
| Pheno] | ND | ND | ND | ND | ND | 900(780) | ~ |
| Methylbenzosulfaamide (p-toluenesulfonamide) | ND | ND | ND | ND | ND | 1000(640) | |
| Benzoic Acid Benzene Carboxylic acid | ND I | ND | ND | ND | ND | 1000(1050) | |
| Dichloroaniline | ND | ND | ND | ND | ND | | 1,290(1000) |

1. See Table 1 for the corresponding EPA sample numbers. ND means none detected, the following limits of detection apply:

chloroaniline
dichloroaniline
chloronitrobenzene
2,4-D
phenol
p-toluenesulfonamide
Benzoic acid
Dichlorophenol

250 ng/mL
500 ng/mL
500 ng/mL
500 ng/mL

| • | $^{\prime\prime}$ |
|---------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| _ | UNITED STATES ENVIRONMENTAL PROTECTION AGENCY |
| DATE: | 5/17/82 m. fut then but, |
| SUBJECT: | Review of Region V Contractor Data; Received for Review on $\frac{5/k/82}{}$ |
| FROM: | Curtis Ross, Director Central Regional Laboratory |
| · TO : | Data User: TAT, CDO, Holos KA |
| | We have reviewed the data for the following Case(s): Site Name: SAUSET SMO Case No: 905 EPA Data Set No: SF140/ Decision Unit: |
| • | CRINO'S: 82WTO6SU/ theu 82WT065U7 |
| • | SMO Traffic No. 's: |
| | Contractor Lab: L'Right State V. Person-hours required for review: |
| | Following are our findings: |
| | note Damples were preserved correctly, but detection limits for this study were higher than the previous study-accounting for the differences in compounds seen/not seen. |
| | |
| | |
| | • |
| | |
| | Data are acceptable for use. but fails to meet program a bjectives Data are unacceptable for use. |
| | () Data are preliminary - this case has been forwarded to Dr. Alfred Haeberer, |
| | EDA Connect Carridge for review - pending reply |

cc: Dr. Alfred Haeberer, EPA Support Services

WRIGHT STATE

May 3, 1982

Wright State University Dayton, Ohio 45435

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|-----------------|----------------|------------------------------|
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| E1208 82WT06S05 | CWS-3 | 1 gallon of water/sediment |
| E1207 82WT06S07 | CWS-4 | 3/4 gallon of water/sediment |
| 82WT06R01 | CWS-5 | 3/4 gallon of water/sediment |

^{1.} Samples were received on January 14, 1982. Samples were packed in styrofoam beads, and ice water was present in shipping containers. Samples CWS-2 and CWS-5 were shipped together in one container and samples CWS-1,-3 and -4 were shipped together in a second container. Caps on bottles were taped.

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ORDER 56606 NAEX

| Con | pound | İs | Listed |
|-----|-------|----|--------|
| in | Task | #2 | 2 |

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- 2. Chloronitrobenzene
- 3. Dichlorophenol
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|-------------------------------------------------|---------|------------------|-------|-------|-------|-------------------------------|------------------------------|
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